

## PATENT ABSTRACTS OF JAPAN

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## (54) ACRYLIC FILM AND ITS LAMINATE

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a film causing little stress-whitening, having high surface hardness and rigidity and excellent in transparency when laminated on a plastic molding or the like.

SOLUTION: This film is obtained by molding a resin composition comprising an acrylic graft copolymer and a methacrylic graft copolymer, wherein the content of acrylic ester-based rubber-like polymer is 5-20 wt.%, the average particle diameter of the acrylic ester-based rubber-like polymer is 500-2,000 Å, and the relation between the average particle diameter d (Å) and the amount w (wt.%) of crosslinking agent used in the acrylic ester-based rubber-like polymer satisfies the formula  $0.002d \leq w \leq 0.005d$ , the graft rate of the acrylic graft copolymer is 30-200% and the reduced viscosity of the methyl ethyl ketone-soluble of the resin composition is 0.2-0.8 dl/g.

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## CLAIMS

[Claim(s)]  
[Claim 1] (A) It is a resin composition which consists of an acrylic graft copolymer containing an acrylic ester system rubber-like polymer and a methacrylic system polymer which contains (B) methyl methacrylate 80% of the weight or more, Content of an acrylic ester system rubber-like polymer (1) 5 to 20 % of the weight, Mean particle diameter of an acrylic ester system rubber-like polymer (2) 500-2000 Å, (3) Relation between mean-particle-diameter [ of the above-mentioned acrylic ester system rubber-like polymer ] d (Å) and the quantity w of a cross linking agent used for an acrylic ester system rubber-like polymer (% of the weight) fills a following formula, 0.002  $d \leq w \leq 0.005d$  dÅ w: A film with which a graft rate of % of the weight (4) acrylic graft copolymer (A) fabricates a resin composition whose reduced viscosity of methyl-ethyl-ketone extractives of 200% and 30 - (5) resin composition is 0.2 - 0.8 dl/g

[Claim 2] A laminated piece which laminated the film according to claim 1.

[Claim 3] An acrylic laminated piece with which the laminated piece according to claim 2 is manufactured by injection molding.

[Translation done.]

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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a specific acrylic film and an acrylic film laminated piece, especially an injection-molded product.

[0002]

[Description of the Prior Art] It is considered as the method of carrying out the decoration of the surfaces, such as plastics and metal goods, and there are a direct printing method and a replica method. However, the direct printing method is unsuitable to the mold goods which have complicated shape, and the technical problem that it was high-cost occurred in the replica method. Methods of giving decoration by low cost include the yne molding method which inserts to an injection molding die, without fabricating, and carries out injection molding of the base material resin, after fabricating films, such as an acrylic resin, by a previous process with vacuum forming etc. Many things are proposed about the acrylic film suitable for this use. For example, the reduced viscosity of a plasticity polymer, the particle diameter of a rubber containing polymer, a rubber content, etc. are indicated by JP.8-323834.A. The reduced viscosity of an acrylic polymer and the content of the multilayer-structure acrylic polymer are indicated by JP.10-279766.A and JP.10-306192.A. These films are excellent in surface hardness, transparency, and a film moldability — the purport statement is carried out. However, nothing is indicated about stress whitening of the film. That is, in this use, when laminating a film to complicated-shaped mold goods, in order that stress may concentrate on a corner etc., these films become easy to milk a film and reduce commodity value remarkably.

[0003]

[Problem(s) to be Solved by the Invention] Then, the multilayer-structure acrylic polymer using the specific acrylic ester system rubber elastomer as a result of inquiring wholeheartedly that this invention persons should develop the film which stress whitening does not produce. The resin composition which consists of an methacrylic system polymer found out that surface hardness is also high few, excel in transparency, and excel in weatherability, and elongation after fracture was also large, and also stress whitening excelled [ elongation after fracture ] also in a film moldability and surface nature, and resulted in this invention.

[0004]

[Means for Solving the Problem] This invention is a resin composition which consists of an acrylic graft copolymer (A) containing an acrylic ester system rubber-like polymer and an methacrylic system polymer (B) which contains methyl methacrylate 80% of the weight or more, Content of an acrylic ester system rubber-like polymer (1) 5 to 20 % of the weight, Particle diameter of an acrylic ester system rubber-like polymer (2) 500-2000 Å, (3) Relation between the particle diameter d of an acrylic ester system rubber-like polymer and the amount w of the amount of cross linking agents used used for an acrylic ester system rubber-like polymer fills a following formula, 0.002  $d < w < 0.005d$  dA w: A graft rate of % of the weight (4) acrylic graft copolymer (A) is an acrylic film which fabricates a resin composition which are \*\*\*\*\* 0.2 of 30 to 200%, and (5) methyl-ethyl-ketone extractives - 0.8 dl/g.

[0005]

[Embodiment of the Invention] The resin composition (G) used for this invention is what consists of an acrylic graft copolymer (A) containing an acrylic ester system rubber-like polymer and an methacrylic system polymer (B) which contains methyl methacrylate 80% of the weight or more, Although acrylic

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graft copolymerization (A) and an methacrylic system polymer (B) are polymerized, respectively and these can be mixed and obtained, after manufacturing an acrylic graft copolymer (A) with the same reaction machine when manufacturing, an methacrylic system polymer (B) can also be continued and manufactured. As a method of mixing, it is mixable with the shape of latex or powder, a bead, a pellet, etc. A krill (meta) acid ester monomer is obtained by the acrylic graft copolymer (A) used for this invention polymerizing under existence of the cross linked rubber-like polymer which used acrylic ester as the main ingredients. An acrylic ester system rubber-like polymer makes the monomeric mixture which consists of a desirable copolymeric cross linking agent of 60 to 99 % of the weight of acrylic ester, 0 to 30 % of the weight of other copolymeric vinyl system monomers, and a specific amount come to polymerize. A monomer may be used, all mixing, and monomer composition may be changed and it may be used in two or more steps. As acrylic ester used here, the thing of the carbon numbers 1-12 of an alkyl group can be used from the point of polymerization nature or cost. As the example, methyl acrylate, ethyl acrylate, acrylic acid propyl, butyl acrylate, 2-ethylhexyl acrylate, acrylic acid n-octyl, etc. are raised, for example, and two or more sorts, however \*\* of these monomers are also good.

[0006] Especially as other copolymerizable vinyl monomers, from the point of weatherability and transparency, methacrylic acid ester is preferred and methyl methacrylate, ethyl methacrylate, methacrylic acid propyl, butyl methacrylate, etc. are raised as the example, for example, Styrene, methylstyrene, etc. are raised as aromatic vinyl and an example of those, and acrylonitrile, a methacrylonitrile, etc. are raised as vinyl cyanides and an example of those. The quantity of a copolymerizable cross linking agent is extended with the mean particle diameter of an acrylic ester system polymer at the time of stress whitening and a fracture, or influences transparency greatly. That is, it is important that the mean particle diameter (dA) and the amount of cross linking agents (% of the weight) of an acrylic ester system elastic body fill a following formula.

0.002  $d < w < 0.005d$ .

[0007] The mean particle diameter of a rubber-like polymer is 500Å - 2000Å. 500Å - 1600 Å (500Å - 1200 Å) are 600Å - 1000Å preferably. In 500Å or less, elongation, shock resistance, etc. fall at the time of a fracture, stress whitening becomes being easy to produce in not less than 2000Å

undesirably, or transparency falls, and it is not desirable.

[0008] The quantity of a cross linking agent has a preferred range shown in the above-mentioned formula, and out of this range, stress whitening arises, elongation falls at the time of a fracture, transparency falls, or a film moldability gets worse, and it is not desirable. Normal use of the cross linking agent used for this purpose is carried out, and often. For example, allyl methacrylate, allyl acrylate, triaryl cyanurate, Triallyl isocyanurate, diallyl phthalate, diallyl maleate, A divinyl horse mackerel peat, divinylbenzene, ethylene glycol dimethacrylate, Diethylene-glycol dimethacrylate, triethylene glycol dimethacrylate, Trimethyl roll propane trimethacrylate, TETROMETHACRYLORUR methanetetra methacrylate, dipropylene glucol dimethacrylate, and these acrylate can be used. Two or more sorts of these cross linking agents may be used.

[0009] An acrylic graft copolymer (A) is obtained by polymerizing the monomer which uses methacrylic acid ester as the main ingredients under existence of said rubber-like polymer. It is obtained from polymerizing 95 to methacrylic-acid-ester 25 weight section preferably in at least one or more steps under existence of said five to acrylic ester system rubber-like polymer 75 weight section. Under the present circumstances, the ingredient which serves as a polymer of a non-graft without carrying out a grafting reaction to an acrylic ester system rubber elastomer arises. This ingredient constitutes some or all of an methacrylic system polymer (B). A graft copolymer becomes insoluble to methyl ethyl ketone. The graft rate over an acrylic ester system rubber elastomer is 30 to 200%, and is 80 to 200% of range preferably. Transparency falls [ a graft rate ] at 30% or less, or elongation falls, undesirably, at not less than 200%, the melt viscosity at the time of film shaping becomes high, a film moldability falls, and it is not desirable. As a monomer used for a grafting reaction, it is methacrylic acid ester, acrylic ester, etc., and is usable in what was used for said acrylic ester system rubber elastomer as an example. As for the using rate of methacrylic acid ester, 50 % of the weight or more is preferred. At 50 or less % of the weight, the hardness and rigidity of a film which are obtained fall and it is not desirable. The methacrylic system polymer (B) used for this invention uses methyl methacrylate as a polymerization component, and contains it 80% of the weight or more. The hardness and rigidity of a film from which methyl methacrylate is obtained at 80 or less

[http://www4.ipdl.inpit.go.jp/cgi-bin/ran\\_web.cgi?atw\\_u=http%3A%2F%2Fwww4.ipdl.i...](http://www4.ipdl.inpit.go.jp/cgi-bin/ran_web.cgi?atw_u=http%3A%2F%2Fwww4.ipdl.i...) 2008/10/30

% of the weight, fall, and it is not desirable.

[0010]As for the content of the acrylic rubber elastomer of the resin composition (C) of this invention, 5 to 20 % of the weight is preferred, and its 10 to 20 % of the weight is more preferred. At 5 or less % of the weight, the elongation of the film obtained does not fall, or it becomes easy to produce stress whitening and is not desirable. At 20 % of the weight or more, the hardness of the film obtained and rigidity fall and it is not desirable. The reduced viscosity of the methyl-ethyl-ketone extractives of the resin composition (C) of this invention is 0.2 – 0.8 dl/g. In 0.2 or less dl/g, the elongation of the film obtained does not fall, or solvent resistance falls, a film moldability falls by 0.8 or more dl/g undesirably, and it is not desirable. The manufacturing method in particular of the acrylic graft copolymer (A) of the resin composition (C) of this invention and an methacrylic system polymer (B) is not limited, and can apply an emulsion polymerization method, a suspension polymerization method, a mass polymerization method, etc.

[0011]The usual polymerization initiator is used in an emulsion polymerization method. As an example, oil-soluble initiators, such as organic peroxide, such as inorganic peroxides, such as potassium persulfate and sodium persulfate, a cumene hydroperoxide, benzoyl peroxide, and also azobisisobutyronitrile, are also used, for example. These are independent, or are combined two or more sorts, and are used. These initiators may be used as a usual redox type polymerization initiator combined with reducing agents, such as sodium sulfite, sodium subsulfite, sodiumformaldehyde, sulfoxylate, ASUKOROBIN acid, and ferrous sulfate. There is no restriction in particular also in the surface-active agent used for said emulsion polymerization, and it can be used if it is a surface-active agent for the usual emulsion polymerizations. For example, nontonic surfactants, such as anionic detergents, such as alkyl-sulfuric-acid soda, alkylbenzene-sulfonic-acid soda, sodium dioctyl sulfosuccinate, and lauric acid soda, and a resultant with alkylphenols and ethyleneoxide, etc. are shown. These surface-active agents may be used independently and may be used together two or more sorts. A resin composition is separated and recovered from the polymer latex obtained with such a copolymer by usual coagulation and washing, or processing by a spray, freezing, etc.

[0012]Especially the acrylic ester system resin composition of this invention is processed good by the tubular film process which is the effective for example, usual melt extruding method as a film, T type die extrusion method or the calendar method, the solvent cast method, etc. About 300–500 micrometers is suitable for the thickness of a film, and its 50–300 micrometers are more preferred.

[0013]It is also possible to obtain the film which is the temperature more than glass transition temperature about a resin composition (C), made film both sides \*\*\*\* simultaneously with a roll or a metal belt if needed, and was superior to that of surface nature.

[0014]The anti-oxidant for raising further the stability to the inorganic matter for coloring of the resin composition (C) of this invention or the paints of an organic system, a color, heat, or light, a thermostabilizer, an ultraviolet ray absorbent, UV stabilizer, etc. — it may be independent, or two or more sorts may be combined, and it may add.

[0015]  
[Example]An example explains this invention below. The “part” in an example and a reference example expresses a weight section, and weight % is expressed “%.” The cable address is as follows.

[0016]OSA: Sodium-dioctyl-sulfosuccinate BA : Butyl-acrylate MMA:methyl-methacrylate GHP:cumene hydroperoxide tDM: Tertiary DODECJIRU mercaptan EA : Ethyl acrylate.

[0017]The characteristic and evaluation followed the following method and conditions.

[0018]Ruthenium staining of the mean-particle-diameter film of an acrylic ester system rubber elastomer was carried out, it observed with the transmission electron microscope, and the particle diameter of 500 pieces was measured.

[0019]Graft rate (G)

It was made to dissolve in methyl ethyl ketone, the powder manufactured by the reference example was divided into insoluble matter and extractives, insoluble matter was considered as a part for a graft, and it asked with the following formula.

[0020]G=(weight of weight/rubber elastomer of weight-rubber elastomer of insoluble matter) x100.

[0021]The reduced viscosity film was dissolved in methyl ethyl ketone, and extractives were measured at 30 \*\* by N,N-dimethylformamide 0.3%. Unit: dl/g.

[0022]The elongation film was pierced to the JIS item type dumbbell at the time of tensile strength and a fracture, and it measured at the speed for \*\*\* speed 50mm/at 23 \*\* at the autograph.

Unit: As for tensile strength, elongation is % at the time of MP and a fracture.

[0023]Cloud value was measured according to JISK6714 using the transparency film. Unit: %.

[0024]According to JISK5400, it measured using the pencil hardness film.

[0025]Film moldability film shaping was performed for 3 hours, the situation was observed, and the next evaluation was carried out.

O : the thickness of a film is uniform and, in the thickness of x:film which can be fabricated without going out, it is generated by unevenness or the film piece.

[0026]The surface of the film of a 1 m of film surface nature <sup>2</sup> size was observed, and the next evaluation was carried out.

O : x:fish eye \*\*:fish eye a fish eye, a die run, and a glow are hardly accepted to be, a die run, or a glow is accepted to be, a die run, or a glow is remarkable.

[0027]The film was bent 180 degrees by 23 \*\* of stress whitening, the white-blush-mark state was observed, and the next evaluation was carried out.

O : x:white blush mark \*\*:white blush mark a white blush mark is not accepted to be is only accepted to be is remarkable.

[0028](Reference example 1) The following substance was taught to 8with agitator L polymerization machine.

[0029]  
Water 200 copies 0.2 copy of OSA Ethylenediaminetetraacetic acid-2-sodium 0.001 copy Ferrous sulfate 0.00025 copy Sodium formaldehyde sulfoxylate After 0.15-copy deoxidization. The mixture (a) shown in Table 1 after the internal temperature was 60 \*\* was continuously dropped at ten copies/hour of a rate, after that, for 30 minutes, postpolymerisation was performed and the acrylic ester system elastic body was obtained. Polymerization conversion was 99.5%. Then, the mixture (b) shown in Table 1 after teaching 0.2 copy of sodium octyl sulfosuccinate was continuously dropped at 12 copies/hour of a rate, postpolymerisation was performed after that for 1 hour, and the acrylic graft copolymer (A) and the methacrylic system polymer (B) were obtained. As for polymerization conversion, the reduced viscosity of the graft rate of methyl-ethyl-ketone extractives was 0.35 dl/g 135% 99.0%. The obtained latex was salted out with calcium acetate, and it solidified, and it rinsed and dried and resin powder was obtained. Reduced viscosity and a graft rate were measured and it was shown in Table 1.

[0030](Reference examples 2-9) The reference examples 2, 3, 4, 5, 6, 7, 8, and 9 as well as the reference example 1 were manufactured by the formula shown in Table 1. Reduced viscosity and a graft rate were measured and it was shown according to Table 1.

[0031]  
[Table 1]

参考例										
	1	2	3	4	5	6	7	8	9	
混合物 ( a )	OSA(部)	0.20	0.15	0.24	0.28	0.26	0.26	0.20	0.05	0.20
	(部)	16	18	14	30	30	30	16	30	16
	BA(%)	90	90	98	90	89	89	90	90	90
	MMA(%)	7.6	7.4	—	7.5	8	7.5	9.0	7.5	8.6
	AIMA(%)	2.4	2.6	2.0	2.5	3	3.5	1.0	2.5	2.4
混合物 ( b )	CHP(部)	0.06	0.06	0.05	0.09	0.09	0.09	0.06	0.09	0.06
	(部)	84	82	86	70	70	70	84	70	84
	BA(%)	10	10	10	10	8	8	10	10	10
	MMA(%)	90	90	90	90	92	9.2	90	90	90
	CHP(部)	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
	tDM(部)	0.28	0.30	0.25	0.25	0.25	0.25	0.28	0.30	0.60
	還元粘度	0.35	0.32	0.38	0.32	0.39	0.39	0.34	0.28	0.15
	グラフト率	135	140	125	120	150	165	110	115	28

(Reference example 10) The copolymer was similarly manufactured using MMA92% and BA8% of monomer by the emulsion polymerization. The reduced viscosity of the obtained methacrylic-acid-ester system copolymer was 0.36 dl/g.

[0032](Reference example 11) The MMA-EA copolymer (the polymer, reduced viscosity 0.30 dl/g which consist of EAabout 5% SUMIPEKKUSU EX;MMAabout 95% by Sumitomo Chemical Co., Ltd.) manufactured by suspension polymerization was used.

[0033](Examples 1-7 and comparative examples 1-5) To 100 copies of resin compositions shown in Table 2 using the obtained polymer, as an ultraviolet ray absorbent, 0.3 copy of Sumi Reiser GM (made by Sumitomo Chemical Co., Ltd.) was mixed as CHIMUBIN 1577 (made by tiba special chemical company) 1.5 copy, and an antioxidant, it extruded at 220 \*\* with the vent type extruder, and the pellet was obtained. The obtained pellet was fabricated with the T-die extruder with the die temperature of 240 \*\*, and the film of 100-micrometer thickness was obtained. Various physical properties were evaluated using this film. The result was shown in Table 2.

[0034]

[Table 2]

	実施例										比較例				
	1	2	3	4	5	6	7	1	2	3	4	5			
参考例-1(純)	100														
参考例-2		100													
参考例-3			100												
参考例-4				60	50			100							
参考例-5						60									
参考例-6							60								
参考例-7								100							
参考例-8									60	10					
参考例-9											90				
参考例-10					50	40	40					40			
参考例-11				40											
高圧体の粒子径	800	1200	800	780	780	800	800	780	800	2500	2500	810			
重量割合	2.4	2.4	2.0	2.5	3.0	3.0	3.5	2.5	1.0	2.5	2.5	2.4			
ゴム量	16	16	14	16	15	18	18	30	16	18	3	16			
グリアム率	135	140	125	120	120	150	105	90	110	115	115	28			
還元粘度	0.35	0.32	0.38	0.31	0.34	0.30	0.32	0.32	0.33	0.29	0.38	0.19			
引張強度	65	61	71	62	68	62	61	32	58	64	90	28			
破断伸び	70	80	60	65	60	55	65	100	60	50	5	1			
応力白化	○	△	○	○	○	○	○	○	○	○	×	×			
透明度	0.3	0.6	0.3	0.4	0.3	0.3	0.3	0.9	0.8	2.3	3.5	2.9			
耐熱強度	H	H	2H	H	H	H	H	B	H	H	3H	HB			
フィルム成形性	○	○	○	○	○	○	○	○	○	○	○	×			
フィルム表面性	○	○	○	○	○	○	○	△	△	△	○	△			

[0035]

[Effect of the Invention]It turns out that the film of this invention is extended at the time of tensile strength and a fracture, and it excels with the sufficient balance of stress whitening, transparency, the degree of surface height, and surface nature.

[Translation done.]

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CORRECTION OR AMENDMENT

[Kind of official gazette]Printing of amendment by the regulation of 2 of Article 17 of Patent Law  
[Section classification] The 3rd classification of the part III gate  
[Publication date]August 14 (2008.8.14), Heisei 20  
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[Written amendment]  
[Filing date]June 26, Heisei 20 (2008.6.26)  
[Amendment 1]  
[Document to be Amended]Specification  
[Item(s) to be Amended]0008  
[Method of Amendment]Change  
[The contents of amendment]  
[0008]

The quantity of a cross linking agent has a preferred range shown in the above-mentioned formula, and out of this range, stress whitening arises, elongation falls at the time of a fracture, transparency falls, or a film moldability gets worse, and it is not desirable. Normal use of the cross linking agent used for this purpose is carried out, and often. For example, allyl methacrylate, allyl acrylate, triaryl cyanurate, Triaryl isocyanurate, diallyl phthalate, diallyl maleate, A divinyl horse mackerel peat, divinylbenzene, ethylene glycol dimethacrylate, Diethylene-glycol dimethacrylate, triethylene glycol dimethacrylate, Trimethyl roll propane trimethacrylate, tetramethylolmethanetetra methacrylate, dipropylene glucocoi dimethacrylate, and these acrylate can be used. Two or more sorts of these

cross linking agents may be used.

[Amendment 2]  
[Document to be Amended]Specification  
[Item(s) to be Amended]0011  
[Method of Amendment]Change  
[The contents of amendment]  
[0011]

The usual polymerization initiator is used in an emulsion polymerization method. As an example, oil-soluble initiators, such as organic peroxide, such as inorganic peroxides, such as potassium persulfate and sodium persulfate, a cumene hydroperoxide, benzoyl peroxide, and also azobisisobutyronitrile, are also used, for example. These are independent, or are combined two or more sorts, and are used. These initiators may be used as a usual redox type polymerization initiator combined with reducing agents, such as sodium sulfite, sodium subsulfite, sodiumformaldehyde sulfoxylate, ASUKOROBIN acid, and ferrous sulfate. There is no restriction in particular also in the surface-active agent used for said emulsion polymerization, and it can be used if it is a surface-active agent for the usual emulsion polymerizations. For example, nonionic surfactants, such as anionic detergents, such as alkyl-sulfuric-acid soda, alkylbenzene-sulfonic-acid soda, sodium dioctyl sulfosuccinate, and lauric acid soda, and a resultant with alkylphenols and ethyleneoxide, etc. are shown. These surface-active agents may be used independently and may be used together two or more sorts. A resin composition is separated and recovered from the polymer latex obtained with such a copolymer by usual coagulation and washing, or processing by a spray, freezing, etc.

[Amendment 3]  
[Document to be Amended]Specification  
[Item(s) to be Amended]0033  
[Method of Amendment]Change  
[The contents of amendment]  
[0033]

(Examples 1-7 and comparative examples 1-5) To 100 copies of resin compositions shown in Table 2 using the obtained polymer, as an ultraviolet ray absorbent, 0.3 copy of Sumi Reiser GM (made by Sumitomo Chemical Co., Ltd.) was mixed as tinuvin\_1577 (made by tiba special chemical company) 1.5 copy, and an antioxidant, it extruded at 220 \*\* with the vent type extruder, and the pellet was obtained. The obtained pellet was fabricated with the T-die extruder with the die temperature of 240 \*\*, and the film of 100-micrometer thickness was obtained. Various physical properties were evaluated using this film. The result was shown in Table 2.

[Translation done.]